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COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

0.21

0.21

FILE 'REGISTRY' ENTERED AT 18:01:26 ON 06 NOV 2007

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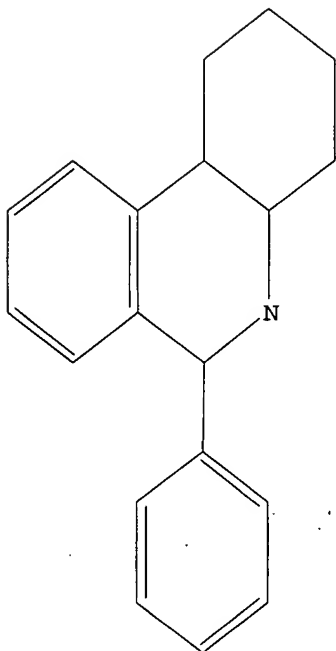
Uploading C:\Program Files\Stnexp\Queries\10524820.str

L1 STRUCTURE UPLOADED

=> d l1

L1 HAS NO ANSWERS

L1 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1

SAMPLE SEARCH INITIATED 18:01:43 FILE 'REGISTRY'

SAMPLE SCREEN SEARCH COMPLETED - 3548 TO ITERATE

56.4% PROCESSED 2000 ITERATIONS  
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)  
SEARCH TIME: 00.00.01

50 ANSWERS

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
BATCH \*\*COMPLETE\*\*

PROJECTED ITERATIONS: 67388 TO 74532

PROJECTED ANSWERS: 2092 TO 3512

L2 50 SEA SSS SAM L1

=>

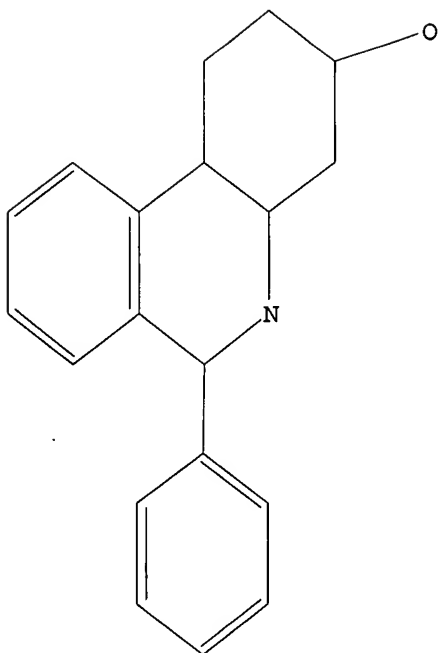
Uploading C:\Program Files\Stnexp\Queries\10524820.str

L3 STRUCTURE UPLOADED

=> d l3

L3 HAS NO ANSWERS

L3 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l3

SAMPLE SEARCH INITIATED 18:03:31 FILE 'REGISTRY'  
 SAMPLE SCREEN SEARCH COMPLETED - 609 TO ITERATE

100.0% PROCESSED 609 ITERATIONS 0 ANSWERS  
 SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
 BATCH \*\*COMPLETE\*\*  
 PROJECTED ITERATIONS: 10700 TO 13660  
 PROJECTED ANSWERS: 0 TO 0

L4 0 SEA SSS SAM L3

=> s l3 ful

FULL SEARCH INITIATED 18:03:42 FILE 'REGISTRY'  
 FULL SCREEN SEARCH COMPLETED - 11864 TO ITERATE

100.0% PROCESSED 11864 ITERATIONS 16 ANSWERS  
 SEARCH TIME: 00.00.01

L5 16 SEA SSS FUL L3

=> file caplus

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

173.45

173.66

FILE 'CAPLUS' ENTERED AT 18:03:45 ON 06 NOV 2007  
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FILE LAST UPDATED: 5 Nov 2007 (20071105/ED)

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=> s 15

L6 12 L5

=> d abs fbib fhitr 1-12

L6 ANSWER 1 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

AB A review of synthetic methods to prepare phenanthridines including cyclization, ring transformation, aromatization and substituent modification. The review includes phenanthridine 5-oxides and phenanthridinium salts.

AN 2005:409854 CAPLUS  
Correction of: 2005:155226

DN 143:248216  
Correction of: 142:197775

TI Product class 11: phenanthridines

AU Keller, P. A.

CS Germany

SO Science of Synthesis (2005), 15, 1065-1088  
CODEN: SSCYJ9

PB Georg Thieme Verlag

DT Journal; General Review

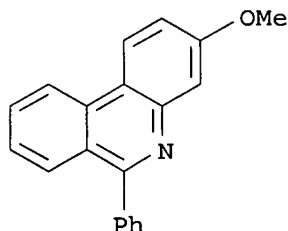
LA English

IT 95128-27-1P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of phenanthridines, phenanthridine-5-oxides and phenanthridinium salts via cyclization, ring transformation, aromatization and substituent modification)

RN 95128-27-1 CAPLUS

CN Phenanthridine, 3-methoxy-6-phenyl- (6CI, 7CI, 9CI) (CA INDEX NAME)



L6 ANSWER 2 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

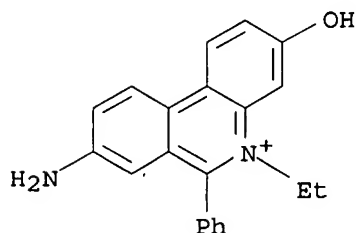
AB Several investigators have described the ultrastructural changes that occur in the mitochondria of cells in tissue cultures after treatment with the drug ethidium bromide (E). It has been assumed that intercalation of E between the base pairs of the mitochondrial DNA induces the formation of the granular inclusions. To investigate whether intercalation is really the initial step in the generation of dense granules inside the matrix, a comparative incubation study of HeLa cell mitochondria was performed in situ with 3 closely related dyes, i.e., E, tetramethylethidium bromide (TME), and betaine B (B). They strongly differ with regard to their affinity for DNA and their ability to cross membranes. E was used as a reference dye. TME does not intercalate, but is externally bound to DNA only weakly. The neutral B is not bound at all, but can cross membranes more easily than the cation E. Moreover, in aqueous solns. at pH 7.0, B is in equilibrium with its protonated cation BH. BH and E have almost equal affinities for DNA. Therefore, B may quickly pass the inner mitochondrial membranes and the cristae, and should then be bound inside the matrix, thus forming a BH-DNA complex. On the assumption that intercalation is necessary for the generation of intramitochondrial electron-dense bodies, it was predicted that BH/B should be more efficient than E, whereas TME should be relatively ineffective. In expts. using HeLa cells, these predictions were found to be inaccurate. E, TME, and BH/B produced almost the same mitochondrial alterations, but at different concns. and after different incubation periods. In contrast to expectations, TME was much more effective than E and BH/B, with the last 2 behaving rather similarly. Therefore, it seems unlikely that the drugs penetrate the inner mitochondrial membrane system by simple phys. diffusion or that intercalation is the preliminary step for the generation of dense granules inside the matrix. Instead, it is assumed that hydrophobic interaction between the dye cations E, BH, and TME and the cristae is the main cause of the mitochondrial changes. The favored binding partner of the dye cations may be the divalent anion, cardiolipin; this phospholipid is an essential part of the inner membrane system but is absent in other membranes of cells. By distributing the dyes between a lipophilic phase and water, it was shown that TME is more lipophilic than E and BH; this may explain the greater effectiveness of TME. The bound dye cations disturb the organization of the cristae, which become altered and finally disappear. It is assumed that the electron-dense granules in the matrix are mainly composed of the dyes and former membrane materials, such as phospholipids and proteins, as well as perhaps some other hydrophobic matrix materials. This would also explain why it was impossible to digest the dense granules by DNase treatment. The drugs enter the mitochondrial matrix by disordering and finally destroying the cristae.

AN 1986:512868 CAPLUS

DN 105:112868

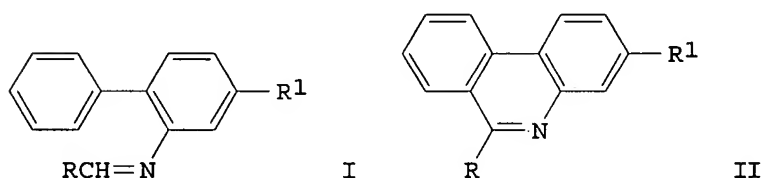
TI Effects of ethidium bromide, tetramethylethidium bromide and betaine B on the ultrastructure of HeLa cell mitochondria in situ. A comparative binding study

AU Roeding, J.; Naujok, A.; Zimmermann, H. W.  
 CS Inst. Phys. Chem., Univ. Freiburg, Freiburg/Br., D-7800, Fed. Rep. Ger.  
 SO Histochemistry (1986), 85(3), 215-22  
 CODEN: HCMYAL; ISSN: 0301-5564  
 DT Journal  
 LA English  
 IT 76357-41-0  
 RL: BIOL (Biological study)  
 (mitochondria of HeLa cell interaction with, ultrastructure in relation to)  
 RN 76357-41-0 CAPLUS  
 CN Phenanthridinium, 8-amino-5-ethyl-3-hydroxy-6-phenyl-, conjugate monoacid (9CI) (CA INDEX NAME)



● H<sup>+</sup>

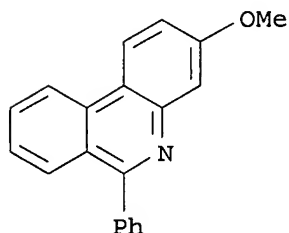
L6 ANSWER 3 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN  
 GI



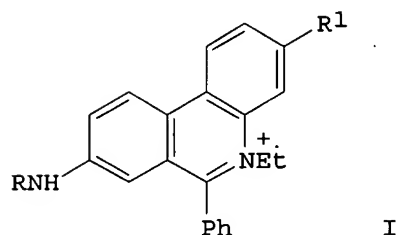
AB The title synthesis builds up the heteroarom. ring via imidoyl radicals chosen because of their ready formation from Schiff bases by hydrogen abstraction. Hydrogen abstraction is performed by treatment with biphenylimines I (R = Ph, p-ClC6H4, p-O2NC6H4, p-anisyl; R1 = H, Cl, OMe) with diisopropyl peroxydicarbonate in benzene at 60°. The formation of imidoyl radicals is followed by intramol. homolytic substitution affording 50-85% 12 phenanthridines II.

AN 1985:541811 CAPLUS  
 DN 103:141811  
 TI A new and convenient synthesis of phenanthridines  
 AU Leardini, Rino; Tundo, Antonio; Zanardi, Giuseppe; Pedulli, Gian Franco  
 CS Ist. Chim. Org., Univ. Bologna, Bologna, I-40136, Italy  
 SO Synthesis (1985), (1), 107-10  
 CODEN: SYNTBF; ISSN: 0039-7881  
 DT Journal

LA English  
OS CASREACT 103:141811  
IT 95128-27-1P  
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
(preparation and spectra of)  
RN 95128-27-1 CAPLUS  
CN Phenanthridine, 3-methoxy-6-phenyl- (6CI, 7CI, 9CI) (CA INDEX NAME)



L6 ANSWER 4 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN  
GI



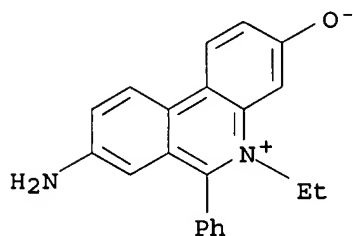
AB Ethidium bromide (I.Br; R = H, R1 = NH2) was acylated to give I (R = Ac, R1 = NH2), which was diazotized, and the diazonium tetrafluoroborate was heated in HOAc containing NaOAc, followed by deacetylation and treatment with alkali to give the title compound I (R = H, R1 = O-)(II). In aqueous solution

II is in a pH-dependent equilibrium with its conjugated aminophenol (I; R = H, R1 = OH)(III) and the dication I (R = H2+, R1 = OH). The pKa of the prototropic equilibrium between II and III at 25° is 7.15. II exhibits a strong neg. solvent effect. Solns. of II in aprotic solvents are blue and in amphiprotic solvents red to purple.

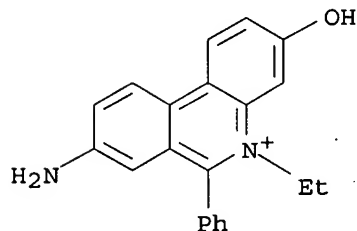
AN 1981:139587 CAPLUS  
DN 94:139587  
TI 8-Amino-5-ethyl-3-oxido-6-phenylphenanthridinium betaine, a new solvatochromic compound  
AU Finkentey, Johann Henrich; Zimmermann, Herbert W.  
CS Inst. Phys. Chem., Univ. Freiburg, Freiburg/Br., D-7800, Fed. Rep. Ger.  
SO Liebigs Annalen der Chemie (1981), (1), 1-6  
CODEN: LACHDL; ISSN: 0170-2041  
DT Journal  
LA German  
IT 77078-15-0P  
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)  
(preparation and color of)  
RN 77078-15-0 CAPLUS



CN Phenanthridinium, 8-amino-5-ethyl-3-hydroxy-6-phenyl-, inner salt (9CI)  
(CA INDEX NAME)

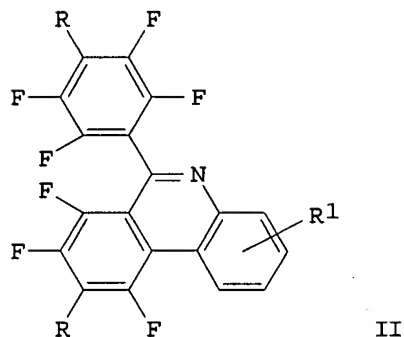


L6 ANSWER 5 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN  
AB Absorption, emission, and polarization spectra of the dication D = BH22+ of betaine B were measured in rigid alc. solution at -195°. D shows besides phosphorescence a delayed fluorescence. The transition moments of fluorescence and phosphorescence are approx. perpendicular.  
AN 1981:55093 CAPLUS  
DN 94:55093  
TI Observation of phosphorescence and delayed fluorescence at the dication BH22+ of betaine B  
AU Finkentey, Johann Henrich; Zimmermann, Herbert W.  
CS Inst. Phys. Chem., Univ. Freiburg, Freiburg/Br., D-7800, Fed. Rep. Ger.  
SO Berichte der Bunsen-Gesellschaft (1980), 84(11), 1133-5  
CODEN: BBPCAX; ISSN: 0005-9021  
DT Journal  
LA German  
IT 76357-41-0  
RL: PRP (Properties)  
(luminescence of)  
RN 76357-41-0 CAPLUS  
CN Phenanthridinium, 8-amino-5-ethyl-3-hydroxy-6-phenyl-, conjugate monoacid (9CI) (CA INDEX NAME)



● H<sup>+</sup>

L6 ANSWER 6 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN  
GI



AB Photochem. cyclization of (p-RC6F4)2C:NC6H4R1 (I; R = F, R1 = H, p-Me, o-Me, p-MeO, m-MeO, o-F; R = CF3, MeO, R1 = H) in CF3CO2H gave 27-85% phenanthridines II. I were obtained in 35-80% yield by treatment of the polyfluoroarom ketones with the corresponding amine.

AN 1981:3905 CAPLUS

DN 94:3905

TI Photochemical cyclization of anils of polyfluoroaromatic ketones

AU Danilenko, N. I.; Fomenko, T. V.; Korobeinicheva, I. K.; Gerasimova, T. N.; Fokin, E. P.

CS Novosib. Inst. Org. Khim., Novosibirsk, USSR

SO Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1980), (7), 1606-11  
CODEN: IASKA6; ISSN: 0002-3353

DT Journal

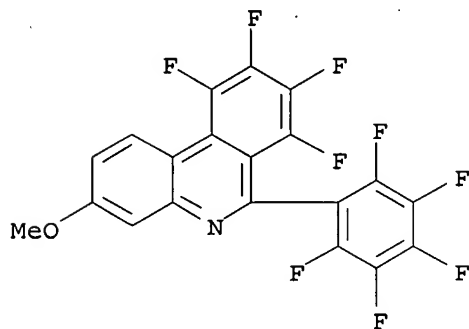
LA Russian

IT 75840-74-3P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of, by photochem. cyclization of anils)

RN 75840-74-3 CAPLUS

CN Phenanthridine, 7,8,9,10-tetrafluoro-3-methoxy-6-(pentafluorophenyl)-  
(9CI) (CA INDEX NAME)



L6 ANSWER 7 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

GI For diagram(s), see printed CA Issue.

AB To a solution of 38 g. Na fluoresceinate and 40 g. NaOH in 1 l. H2O was added 40 g. H2NOH.HCl in 100 ml. H2O, the mixture was left 5 hrs. at 75-80°, made slightly acid with H2SO4, and the resulting precipitate was

washed with Me<sub>2</sub>CO and purified, by precipitation from HCl solution with NaOAc, to

yield fluorescein oxime formulated as the zwitterion of 3,7-dihydroxy-11-(2-carboxyphenyl)dibenz[b,f]oxazepine (I), C<sub>20</sub>H<sub>13</sub>NO<sub>5</sub>; acetyl derivative m. 144°. I on esterification with MeOH in the presence of H<sub>2</sub>SO<sub>4</sub> afforded the Me ester (II) of I; its trimethyl ether was isolated in 400 mg. yield when a mixture of Et<sub>2</sub>O, tetrahydrofuran, and 0.75 g. I was treated 24 hrs. with 0.5 g. CH<sub>2</sub>N<sub>2</sub> and the product was purified on an alumina column. The same compound was obtained from II and CH<sub>2</sub>N<sub>2</sub>. I (2.5 g.) heated 24 hrs. at 200° with 0.5 g. Cu bronze (III) in 25 ml. quinoline (IV), the product extracted with Et<sub>2</sub>O, and worked up, gave 3,7-dihydroxy-11-phenyldibenz[b,f]oxazepine (V); hydrochloride m. 157°. An ethereal solution of V treated 24 hrs. with an excess of CH<sub>2</sub>N<sub>2</sub> gave the 3,7-dimethoxy derivative, m. 132° (alc.). The K salt of 2,4-HO (MeO)C<sub>6</sub>H<sub>3</sub>OMe (5 g.) was heated 24 hrs. at 185° with 4.35 g. 3,4-Br(O<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>OMe and 100 mg. III, the mixture was extracted with

Et<sub>2</sub>O-aqueous NaOH

solution, the organic layer was evaporated, and the residue was fractionated over

silica gel with 1:1 ligroine-Et<sub>2</sub>O to yield 2.75 g. 4-methoxy-2-(2-nitro-5-methoxyphenoxy)benzophenone (VI), m. 124.5-5.5° (alc.). VI (400 mg.), 0.1 ml. NH<sub>2</sub>NH<sub>2</sub>.H<sub>2</sub>O, and Raney Ni was refluxed 0.5 hr. in 10 ml.

EtOH, the mixture was concentrated in vacuo, and the residue was extracted with Et<sub>2</sub>O

and HCl. The aqueous layer was made alkaline, and extracted with Et<sub>2</sub>O to yield 270

mg. hydrochloride, m. 157°, identical with that obtained from V. On the basis of these results and the ir and N.M.R. spectra of I and its derivs., the compound was formulated as 3,7 - dihydroxy - 11- (2' - carboxyphenyl)dibenz[b,f]oxazepine. The α- (VII), β- (VIII), and γ- (IX) oximes of hydroquinonephthalein (X) were prepared by the method of Meyer and Spengler (Ber. 36, 2961 (1903)). VII on acid hydrolysis afforded X and its trimethyl ether on hydrolysis gave the dimethyl ether of X. VII did not rearrange on treatment with PCl<sub>5</sub> and was formulated as spiro[(2-hydroxy-3-oxo-2,3-dihydroisoindol) - 1,9' - (2,7-dihydroxyanthrene)]. IX (0.5 g.), reduced in 150 ml. 0.5N NaOH at -1.65 v. vs. S.C.E., consumed 2 electrons per mol. Reoxidn. of the product afforded IX. Similar electrolytic reduction of VIII followed by oxidation gave IX. Methylation of 400 mg. IX with CH<sub>2</sub>N<sub>2</sub> afforded 300 mg. 2,7-dimethoxy-9-(2-carbomethoxyphenyl)acridine (XI), m. 166°. Methylation of 4 g. VIII yielded 0.5 g. XI and 2.5 g. 2,7-dimethoxy-9-(2-carbomethoxyphenyl)-acridine N-oxide (XII), m. 206°.

Decarboxylation of 0.4 g. XII with 0.2 g. III in 10 ml. IV furnished 120 mg. 2,7-dimethoxy-9-phenylacridine, m. 191°, also obtained in 150 mg. yield on decarboxylation of XI, and in 2.5 g. yield when 10 g. PhCH<sub>2</sub>CHO, 16 g. p-MeOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>, and 16 g. p-MeOC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>.HCl were heated 1 hr. at 220° and the mixture then oxidized with FeCl<sub>3</sub>. The trimethyl ether of IX was oxidized by m-ClC<sub>6</sub>H<sub>4</sub>CO<sub>3</sub>H to the trimethyl ether of VIII. IX was obtained in 1.85 g. yield when 10 g. X was heated 1 hr. on a water bath with 100 ml. 2N NaOH and 50 ml. concentrated aqueous NH<sub>3</sub>. These

observations,

together with N.M.R., ir, and uv spectral data (reported) suggest that IX is 2,7-dihydroxy-9-(2-carboxyphenyl)acridine in form of a zwitterion and that VIII is the corresponding N-oxide. 4,4'-Dimethoxy-2-nitrobiphenyl (XIII), m. 138° (MeOH), was obtained in 8.5 g. yield when 14.3 g.

3,4-O<sub>2</sub>NBrC<sub>6</sub>H<sub>3</sub>OMe and 14.5 g. p-IC<sub>6</sub>H<sub>4</sub>OMe, 120 ml. PhNO<sub>2</sub>, and 80 g. III were refluxed 4 hrs.; the mixture was extracted with CHCl<sub>3</sub>, and the extract purified on

an alumina column. XIII (2 g.), 30 ml. MePh, 13 ml. EtOH, and 2.5 ml.

NH<sub>2</sub>NH<sub>2</sub>.H<sub>2</sub>O heated with 100 mg. Raney Ni gave 79% 4,4'-dimethoxy-2-aminobiphenyl, m. 110° (alc.), and this stirred 1.5 hrs. at room temperature with 3 ml. C<sub>5</sub>H<sub>5</sub>N, and 1 ml. BzCl gave on workup 80% 4,4'-dimethoxy-2-benzamidobiphenyl (XIV), m. 114° (alc.). XIV (200 mg.) refluxed 3 hrs. with 5 ml. POCl<sub>3</sub> and the mixture poured into ice-water gave 120 mg. 3,8-dimethoxy-6-phenylacridine, m. 131° (MeOH). (2,7-Dimethoxyfluorenone (100 mg.) heated 1 hr. at 165° with 1 ml. PhNH<sub>2</sub> and 20 mg. ZnCl<sub>2</sub> and the mixture fractionated on an alumina column gave 50 mg. 2,7-dimethoxyfluorenone anil, m. 121°.

AN 1967:10838 CAPLUS

DN 66:10838

OREF 66:2091a,2094a

TI Constitution of fluorescein oxime and the hydroquinonephthalein oximes

AU Lund, Henning; Lunde, Per; Kaufmann, Frantz

CS Univ. Aarhus, Aarhus, Den.

SO Acta Chemica Scandinavica (1947-1973) (1966), 20(6), 1631-44

CODEN: ACSAA4; ISSN: 0001-5393

DT Journal

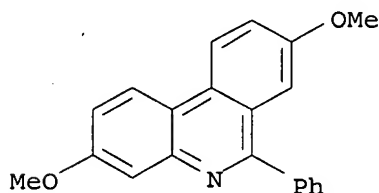
LA English

IT 13606-09-2P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 13606-09-2 CAPLUS

CN Phenanthridine, 3,8-dimethoxy-6-phenyl- (8CI) (CA INDEX NAME)

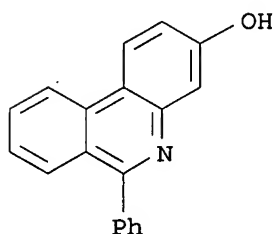


L6 ANSWER 8 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

AB 4-Methyl-2-nitrobiphenyl was prepared by diazotization of 76 g. 4-amino-3-nitrotoluene in 150 ml. concentrated HCl and 100 ml. H<sub>2</sub>O at 0-5° during 1 hr. with 38 g. NaNO<sub>2</sub>. The cold, filtered diazo solution was added rapidly to 1 l. C<sub>6</sub>H<sub>6</sub> at 5° and 160 g. NaOAc in 400 ml. water was added and the mixture stirred 3 hrs. at 305°. The C<sub>6</sub>H<sub>6</sub> was removed in vacuo and the fraction b11 180-200° collected and redistd. 4-Methyl-2-nitrobiphenyl separated from ligroine in pale yellow cubes (50% yield), m. 49°, b11 188-90°. The following biphenyls were similarly prepared (substituents, form, m.p., and % yield given): 2-amino-4-methyl, oil, -- (b11 170-1°), 80; 2-acetamido-4-methyl, plates, 151°, 90; 2-benzamido-4-methyl, needles, 94°, 85; 2-ethoxycarbonylamino-4-methyl, cubes, 73-4°, 65; 4-methoxy-2-nitro, yellow laths, 73-4°, 20; 2-amino-4-methoxy, oil, -- (b12 212-13°), 77; 2-acetamido-4-methoxy, needles, 96-7°, 85; 2-benzamido-4-methoxy, needles, 148°, 80; 4-chloro-2-nitro, yellow cubes, 53°, 51; 2-amino-4-chloro, oil, -- (b12 185-6°), 88; 2-acetamido-4-chloro, prisms, 129.5°, 75; 2-benzamido-4-chloro, prisms, 104-5°, 75; 4-chloro-2-ethoxycarbonylamino, cubes, 130-1°, 70; 2-ethoxycarbonylamino, needles, 186°, 70; 2-amino-4'-ethoxycarbonylamino, plates, 98°, 70; 2-amino-4'-benzamido, needles, 180-1°, 80; 2-acetamido-3,5-dibromo, rhombohedrons,

134-5°, 78. 2-Acetamido-4-methylbi-phenyl (5 g.) and 15 ml. POCl<sub>3</sub> were refluxed 2 hrs. until evolution of HCl practically ceased. The excess POCl<sub>3</sub> was removed in vacuo and the residue poured on 200 g. ice and neutralized with aqueous NH<sub>3</sub> to give 75% 3,6-dimethylphenanthridine, m. 105° (petroleum); picrate m. 255° (decomposition) (EtOH). The following phenanthridines were prepared similarly (substituents, m.p., picrate, m.p., and % yield given): 3-methyl-6-phenyl, 118.5°, 259° (decomposition), 80; 3-methoxy-6-methyl, 85°, 248° (decomposition), 65; 3-methoxy-6-phenyl, 115.5°, 267° (decomposition), 70; 3-hydroxy-6-methyl, 295-6° (decomposition), --, 75; 3-benzoyloxy-6-methyl, 1489°, --, 85; 3-hydroxy-6-phenyl, 297-8° (decomposition), --, 85; 3-benzoyloxy-6-phenyl, 191-2°, --, 80; 3-chloro-6-methyl, 131.5-32°, 233° (decomposition), 65; 3-chloro-6-phenyl, 142.5°, 257° (decomposition), 70; 2-bromo-6-methyl, 132°, 245° (decomposition), 60; 2,4-dibromo-6-methyl, 173-4°, 201° (decomposition), 70; 2,4-dibromo-6-phenyl, 208°, --, 60.

AN 1962:12942 CAPLUS  
 DN 56:12942  
 OREF 56:2424d-h  
 TI Some derivatives of biphenyl and of phenanthridine  
 AU Hollingsworth, B. L.; Petrow, U.  
 CS Univ. London  
 SO Journal of the Chemical Society (1961) 3771-3  
 CODEN: JCSOA9; ISSN: 0368-1769  
 DT Journal  
 LA Unavailable  
 OS CASREACT 56:12942  
 IT 94210-65-8P, 3-Phenanthridinol, 6-phenyl-  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 94210-65-8 CAPLUS  
 CN 3-Phenanthridinol, 6-phenyl- (6CI, 7CI) (CA INDEX NAME)



L6 ANSWER 9 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN  
 AB cf. C.A. 52, 20193b. 11-Phenyldibenzo[b,f]-1,4-thiazepines variously substituted in the 2- and (or) 8-position were synthesized and the effects of these substituents on extrusion of S examined 2-Benzamidodiaryl sulfide (1 mole), 4 moles POCl<sub>3</sub>, and 13 moles PhNO<sub>2</sub> refluxed 4 hrs., the volatiles removed at 0.1 mm., and the residue triturated with dilute NaOH gave the 11-phenyldibenzo [b,f]-1,4-thiazepine (I). By-products were obtained in 2 cases. Methods A and B. The usual trituration of the crude product from 2-benzamido-4-methoxydiphenyl sulfide gave a considerable amount of yellow solid. This solid heated 0.5 hr. with dilute aqueous NaOH gave 2-methoxy-9-phenylphenanthridine; alternatively a hot solution of the original solid in dilute H<sub>2</sub>SO<sub>4</sub> gave needles, m. 283° (decomposition), the sulfate of the same base. The C<sub>6</sub>H<sub>6</sub> extract of the crude product from

2-benzamido-4'-methoxy-4-nitrodiphenyl sulfide gave a small crop of 5-nitro-2-phenylbenzothiazole, m. 138° (alc.). This compound was apparently formed by scission of the diaryl sulfide and cyclization of the resultant 2-benzamido-4-nitrothiophenol. Method C. 4,4'-Dimethyl-2,2'-dinitrodiphenyl disulfide in AcOH at 100° treated with successive addns. of concentrated HCl and Zn dust, the hot filtered solution added to H<sub>2</sub>O, the

Zn salt dried, and dissolved in concentrated HCl crystallized 2-amino-4-methylthiophenol-HCl, m. 189° (decomposition). It was condensed with 2-chloro-5-nitrobenzophenone as described for the lower homolog. 2- and 8-Hydroxythiazepine were obtained by demethylating 2- and 8-methoxythiazepine with HBr in AcOH 3 hrs. under reflux. The following I were thus prepared (2 and 8 substituents, method, m.p. given): H, H, A, 118°; H, Cl, A, 150°; Cl, H, A, 134°; H, Me, A, 127°; Me, H, A, 164°; H, MeO, A, 159°; MeO, H, A, 140°; H, OH, -, 255°; OH, H, -, 304°; H, NO<sub>2</sub>, B, 187°; NO<sub>2</sub>, H, C, 159°; Cl, Cl, A, 152°; Me, Cl, A, 147°; Me, NO<sub>2</sub>, B, 187°; NO<sub>2</sub>, Me, C, 177°; MeO, NO<sub>2</sub>, B, 138°. The 2-nitrodiaryl sulfides were prepared by dropwise addition of 30% NaOH to a solution of the appropriate thiophenol and o-ClC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub> in 500 cc. alc. The whole being heated 0.5 hr. and the product crystallized from MeOH or alc. The following 4- and/or 4'-substituted 2-nitrodiphenyl sulfides were thus prepared (4', 4 substituents, and m.p. given): H, H, 79°; H, Cl, 84°; Cl, H, 97°; H, Me, 72°; Me, H, 89°; H, MeO, oil; MeO, H, 94°; Cl, Cl, 158°; Me, Cl, 121°. 2-Nitrodiaryl sulfides were reduced to the amines which were isolated as crude HCl salts and then treated with BzCl at room temperature 2-3 hrs. in C<sub>5</sub>H<sub>5</sub>N. After addition

of dilute H<sub>2</sub>SO<sub>4</sub> and recovery from alkali washed and dried Et<sub>2</sub>O exts., all except the last 3 compds. were thus obtained. These 3 compds. were prepared by the procedure for 2-nitrodiaryl sulfides but using 2-chloro-5-nitrobenzanilide in place of the ClC<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>. The following 4- and (or) 4'-substituted 2-benzamidodiphenyl sulfides were obtained (4'- and 4-substituents and m.p. given): H, H, 67°; H, Cl, 81°; Cl, H, 102°; H, Me, 91°; Me, H, 94°; H, MeO, 61°; MeO, H, 78°; Cl, Cl, 110°; Me, Cl, 85°; H, NO<sub>2</sub>, 115°; Me, NO<sub>2</sub>, 125°; MeO, NO<sub>2</sub>, 142°. I (0.001 mole), 0.4 g. Cu bronze, and 3 cc. di-Et phthalate under N was plunged into a metal bath at 315°, after the stated time the flask removed, its contents cooled, transferred, and refluxed with C<sub>6</sub>H<sub>6</sub>, the suspension filtered through C, and the filtrate either concentrated to 25 cc. and treated with saturated picric acid in C<sub>6</sub>H<sub>6</sub> and the picrate collected after 0.5 hr., or when picrate precipitation was incomplete even after 12 hrs., the solvents removed

and the bases fractionally crystallized from alc. 9-Phenylphenanthridines (II) liberated from the picrates by treatment with alkali were recovered in C<sub>6</sub>H<sub>6</sub>. The following II were obtained (2- and 8-substituents, m.p., and m.p. of picrate given): H, H, 105°, 251°; H, Cl, 134°, 245°; Cl, H, 120°, 260°; H, Me, 119°, 245°; Me, H, 90°, 280°; H, MeO, 116°, 260°; MeO, H, oil, 270°; H, OH, 290°, 295°; OH, H, 266°, 245°; H, NO<sub>2</sub>, 223°, 215°; NO<sub>2</sub>, H, 236°, 247°; Cl, Cl, 196°, 214°; Me, Cl, 179°, 232°; Me, NO<sub>2</sub>, 228°, 209°; NO<sub>2</sub>, Me, 229°, 213°; MeO, N<sub>2</sub>O, 213°, 231°. Reactions in quinoline were similarly conducted, solvents being removed in vacuo, and the residue in C<sub>6</sub>H<sub>6</sub> washed with 1.5N AcOH. II were precipitated as picrates and thereafter unchanged thiazepines recovered from

the alkali-washed C6H6 mother liquors. Thus the conversion of I to II was carried out (2- and 8-substituents, % yield after 60 min. at 240°, % yield at 10 min., 15 min., and 0.5 hr. at 300° given): H, H, nil, 57, 73, 86; H, Cl, -, 61, 73, 81; Cl, H, -, 56, 71, 84; H, Me, 38, 80, 85, 86; Me, H, -, 51, -, 83; H, MeO, 79, 82, -, 82; MeO, H, 10, 77, 85, 88; H, OH, 76, 75, -, -; OH, H, nil, (52), -, -; H, NO2, -, nil, -, 15°; H, 30, 79, -, -; Cl, Cl, -, 65, 77, 83; Me, Cl, -, 61, 79, 86; Me, NO2, -, nil, -, 20; NO2, Me, 60, 88, -, -; MeO, NO2, 9, 60, -, -.

AN 1959:99900 CAPLUS

DN 53:99900

OREF 53:18056a-i,18057a-b

TI Extrusion of sulfur. IV. Effect of substituents in 11-phenyldibenzo[b,f]-1,4-thiazepine

AU Galt, R. H. B.; Loudon, J. D.

CS Univ. Glasgow, UK

SO Journal of the Chemical Society (1959) 885-9

CODEN: JCSOA9; ISSN: 0368-1769

DT Journal

LA Unavailable

IT 95953-09-6

RL: PREP (Preparation)

(Derived from data in the 6th Collective Formula Index (1957-1961))

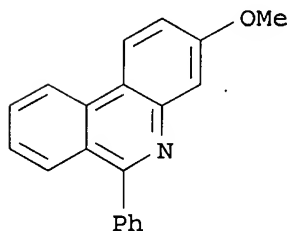
RN 95953-09-6 CAPLUS

CN Phenanthridine, 3-methoxy-6-phenyl-, picrate (6CI, 7CI) (CA INDEX NAME)

CM 1

CRN 95128-27-1

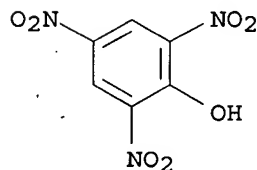
CMF C20 H15 N O



CM 2

CRN 88-89-1

CMF C6 H3 N3 O7



L6 ANSWER 10 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

AB 3,5-Dialkyl-4-nitrosopyrazole treated with PCl5 yielded the corresponding

3,6-dialkyl-5-chloro-1,2,4-triazine. By analogy to the known reaction of  $\text{PCl}_5$  with isonitroso ketones it was postulated that this reaction proceeded through the 1,4-dialkyl-1-chloro-4-cyano-2,3-diaza-1,3-butadiene,  $\text{R}'\text{C}(\text{Cl})\text{:NN:C}(\text{CN})\text{R}$  (I), a new class of compds. In some cases it was possible to sep. both isomers of (I) and determine their structures. I treated with  $\text{NH}_3$  yielded the corresponding 3,6-dialkyl-5-amino-1,2,4-triazine which was then converted to the OH, Cl,  $\text{NHNH}_2$  or H derivative 3-Methyl-4-isonitroso-4-pyrazolone treated with  $\text{PCl}_5$  yielded 2-(chloroformylhydrazyl)propionitrile,  $\text{ClCONHN:C}(\text{CN})\text{Me}$ . The latter yielded the expected semicarbazone with  $\text{NH}_3$  or amines and a sym. carbohydrazone with water.

AN 1959:99899 CAPLUS

DN 53:99899

OREF 53:18055h-i,18056a

TI A new pyrazole ring-opening reaction: New syntheses of asymmetric triazines

AU Fusco, Raffaello; Rossi, Silvano

CS Univ. Milan

SO Atti accad. nzal. Lincei, Rend., Classe sci. fis., mat e nat. (1956), 21, 208-210

DT Journal

LA Unavailable

IT 95953-09-6

(Derived from data in the 6th Collective Formula Index (1957-1961))

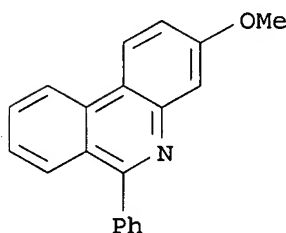
RN 95953-09-6 CAPLUS

CN Phenanthridine, 3-methoxy-6-phenyl-, picrate (6CI, 7CI) (CA INDEX NAME)

CM 1

CRN 95128-27-1

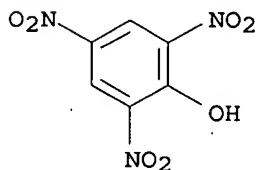
CMF C20 H15 N O



CM 2

CRN 88-89-1

CMF C6 H3 N3 O7





**REMARKS**

Upon entry of the above amendment, claims 1-12, 14 and 17-19 will be pending in this application. Claims 1-12, 14 and 17-18 have been amended to include hydrates, solvates, salts, hydrates of the salts and solvates of the compounds of formula I. Specific basis for this amendment can be found at page 5, 5<sup>th</sup> paragraph. Further, the claims have been amended to recite the singular "A compound" in lieu of the plural "Compounds".

Neither the amendments to the claims nor the addition of new claim 19 introduce new matter within the meaning of 35 U.S.C. §132. Accordingly, the Examiner is respectfully requested to enter the above amendment before examination.

If the Examiner has any questions regarding this submission, she is invited to telephone the undersigned attorney.

Respectfully submitted,  
NATH & ASSOCIATES PLLC

By: \_\_\_\_\_

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Registration No. 26,965  
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L6 ANSWER 11 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

AB The observation that a 6-phenylphenanthridinium salt with only MeO as substituent possessed significant trypanocidal properties suggested the examination of a series of such compds. and of similar compds. with HO groups. 4-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>-NHAc-2 (42 g.) in 240 mL. N H<sub>2</sub>SO<sub>4</sub>, diazotized with 17 g. NaNO<sub>2</sub> (with addition of 240 mL. N H<sub>2</sub>SO<sub>4</sub>) and the diazonium solution decomposed under PhMe (48 h. at 0°), gives 35 g. 2-acetamido-4'-hydroxybiphenyl (I), m. 185-6°. I (61 g.) in 274 mL. N NaOH, heated on a steam bath and treated gradually with 29 mL. Me<sub>2</sub>SO<sub>4</sub>, give 50 g. 2-acetamido-4'-methoxybiphenyl (II), b0.01 130°, m. 134°; the following 4'-alkoxy derivs. were similarly prepared: EtO, b0.01135°, m. 91°; iso-PrO, m. 108-9.5°; BuO, b. 155°/1 + 10-5 mm., m. 98°; PhCH<sub>2</sub>O, b. 190-200°/1 + 10-5 mm., m. 138°; ethylcarbonato, m. 127-8°; BzO, m. 187-8°. 2-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>NH<sub>2</sub>-4 (32.5 g.) yields 22 g. 2-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>OH-4, m. 110-12°; acetate, m. 122°; Me ether, m. 60-60.5°; Et ether, m. 51°; Et carbonate, m. 121°. 2-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>OMe-4 on catalytic reduction over 5% Pd-C at 70°/50 atmospheric gives a nearly quant. yield of 2-amino-4'-methoxybiphenyl (III), b0.11 118-20°, m. 36°; HCl salt, m. 227-8°. 2-Amino-4'-ethoxybiphenyl, b0.1 130-5°, m. 56°; 4'-(ethylcarbonato) analog, m. 65-6°. II (50 g.) in 24 mL. concentrated H<sub>2</sub>SO<sub>4</sub> and 450 mL. EtOH, refluxed 1.5 h., gives 31 g. III; 2-amino-4'-isopropoxybiphenyl, b0.05 128-34°; BuO homolog, b0.003 140°; benzyloxy analog, m. 122°. 3,4-O<sub>2</sub>N(H<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>OMe (150 g.) in 280 mL. hot 10 N HCl and 75 mL. H<sub>2</sub>O, diazotized at 0° with 67.5 g. NaNO<sub>2</sub> and stirred vigorously with 2 l. C<sub>6</sub>H<sub>6</sub> at 5-10° while being treated with 300 g. AcONa in concentrated aqueous solution, gives (after 48 h.)

2-nitro-4-methoxy-biphenyl, pale yellow, b0.05 120-35°, m. 75-7°; catalytic reduction gives 2-amino-4-methoxybiphenyl, b0.08 128-30°. p-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H and ClCO<sub>2</sub>Et with an equivalent quantity of PhNEt<sub>2</sub> (or the HCl salt of the acid and excess ClCO<sub>2</sub>Et) give p-EtO<sub>2</sub>CNHC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H, m. 203° the acid chloride m. 110°. The following o-acylaminobiphenyls were prepared from 0.1 mol. 2-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>OR-4 in 200 mL. CHCl<sub>3</sub> and 16 g. Na<sub>2</sub>CO<sub>3</sub> by treatment with 10% excess R'COCl, with stirring for 30 min. and refluxing a further 30 min. 2-XC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>OMe-4 (X given): BzNH, m. 108°; p-MeOC<sub>6</sub>H<sub>4</sub>CONH, m. 146°; p-ClC<sub>6</sub>H<sub>4</sub>CONH, m. 142-3°; p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CONH, m. 164-5°; m-isomer, m. 136.5-7°; p-EtO<sub>2</sub>CNHC<sub>6</sub>H<sub>4</sub>CONH, m. 178°; 3,5-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CONH, m. 179-80.5°; p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CONH, m. 149-50°. 2-(p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>CONH) C<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>X-4 (X given): EtO, m. 147°; iso-PrO, m. 139°; BuO, m. 118°; PhCH<sub>2</sub>O, m. 146-7°. 2-(Phenylacetamido)biphenyl, m. 85-6°; 2-[α-(p-methoxyphenyl)acetamido]biphenyl, m. 105°. Phenanthridines were prepared from o-(acylamino)biphenyls in POCl<sub>3</sub> (1 mL. to 1 g. amide) by refluxing 5 h., grinding with CHCl<sub>3</sub> and concentrated HCl, and treating with NH<sub>4</sub>OH; salts were prepared by heating the phenanthridines 15 min. at 150° with Me<sub>2</sub>SO<sub>4</sub> and transformed into the chlorides with concentrated HCl. The NH<sub>2</sub> compds. were prepared from the NO<sub>2</sub> compds. by reduction with Fe powder or Fe(OH)<sub>2</sub>; demethylation was effected by heating with concentrated HCl

h. at 160-70°. Phenanthridines (C.A. numbering): 8-methoxy-6-Me, b0.003 141-8°, m. 57°, 60%; 8-(ethylcarbonato)-6-Me, m. 98-9°, variable yield (HCl salt, m. 205-6°); 8-benzyloxy-6-Me, m. 208-9°, 20%; 8-methoxy-6-Ph, b0.01 190-2°, 67% (picrate, m. 270-1°); 8-methoxy-6-(p-methoxyphenyl), m. 94°, 80%; 8-methoxy-6-(p-chlorophenyl), m. 157-8°, 73%; 8-methoxy-6-(p-nitrophenyl), m. 233-4°, 72%; 8-methoxy-6-(m-nitrophenyl), m.

183-4°, 81%; 8-methoxy-6-(p-carbethoxyaminophenyl), m. 132-6°, 70%; 8-methoxy-6-(3,5-dinitrophenyl), m. 252-3°, 57%; 8-ethoxy-6-(p-nitrophenyl), m. 233°, 60%; 8-propoxy-6-(p-nitrophenyl), m. 169°, 60%; 8-propoxy-6-(m-nitrophenyl), m. 146°, 60%; 8-isopropoxy-6-(p-nitrophenyl), m. 159-60°, 60%; 8-butoxy-6-(p-nitrophenyl), m. 173°, 67%; 8-benzyloxy-6-(p-nitrophenyl), m. 180°, 70%; 3-methoxy-6-(p-nitrophenyl), m. 199°, 90%; 6-(p-methoxybenzyl), m. 125-7°, 70%; 8-hydroxy-6-Me, m. 298-300°, 95% (HCl salt, m. 285-7°); 8-hydroxy-6-(p-nitrophenyl), m. 275°, 80%; 8-hydroxy-6-(m-nitrophenyl), m. 250°, 75%. Alkoxy-5-methylphenanthridinium chlorides: 8-methoxy-6-Me (bromide), m. 238°; 8-methoxy-6-Ph, m. 216-17°; 8-methoxy-6-(p-methoxyphenyl), m. 215-16°; 8-methoxy-6-(p-chlorophenyl), m. 229-30°; 8-methoxy-6-(p-nitrophenyl), m. 230-2°; 8-methoxy-6-(p-aminophenyl), m. 253-4° (Ac derivative, m. 258-9°); 8-methoxy-6-(p-carbethoxyaminophenyl), m. 248-9°; 8-methoxy-6-(m-nitrophenyl), m. 229°; 8-methoxy-6-(m-aminophenyl), m. 236-8°; 8-methoxy-6-(m-carbethoxyaminophenyl), m. 232-3°; 8-methoxy-6-(3,5-dinitrophenyl), m. 260°; 8-methoxy-6-(3,5-diaminophenyl), m. 232-3°; 8-ethoxy-6-(p-nitrophenyl), m. 233°; 8-ethoxy-6-(p-aminophenyl), m. 225-6°; 8-propoxy-6-(p-nitrophenyl), m. 222°; 8-propoxy-6-(p-aminophenyl) (IV), m. 215°; 8-propoxy-6-(m-nitrophenyl), m. 212°; 8-propoxy-6-(m-aminophenyl), m. 206°; 8-propoxy-6-(m-carbethoxyaminophenyl), m. 207°; 8-isopropoxy-6-(p-nitrophenyl), m. 239-40°; 8-isopropoxy-6-(p-aminophenyl), with 2 mols. H<sub>2</sub>O, m. 174-5°; 8-butoxy-6-(p-nitrophenyl), m. 234°; 8-butoxy-6-(p-aminophenyl), m. 190°; 8-benzyloxy-6-(p-nitrophenyl), m. 226°; 8-benzyloxy-6-(p-aminophenyl), m. 184°; 3-methoxy-6-(p-nitrophenyl), m. 234°; 3-methoxy-6-(p-aminophenyl), m. 235°. 8-Hydroxy-5-methylphenanthridinium chlorides: 6-Me, m. 262-3°; 6-Ph, m. 263-4°; 6-(p-hydroxyphenyl), m. 264-5°; 6-(p-chlorophenyl), m. 244-5°; 6-(p-nitrophenyl), m. 252°; 6-(p-aminophenyl), m. 269-71°; 6-(p-carbethoxyaminophenyl), m. 281-2°; 6-(m-aminophenyl), m. 275-7°; 6-(m-carbethoxyaminophenyl), m. 198-200°. 3-Hydroxy-6-(p-nitrophenyl)-5-methylphenanthridinium chloride, m. 330°; 6-(p-aminophenyl) analog, m. 297-8°. 5-Methylphenanthridinium chlorides: 6-benzyl, m. 205-7°; 6-(p-methoxybenzyl), m. 192°; 8-methoxy-6-(p-nitrobenzyl), m. 151°; 8-methoxy-6-(p-aminobenzyl), m. 241°; 8-methoxy-6-(p-carbethoxyaminobenzyl) (V), m. 220°; 8-methoxy-6-(p-propionylaminobenzyl), m. 218°; 8-hydroxy-6-(p-aminobenzyl), m. 252°; 8-hydroxy-6-(p-carbethoxyaminobenzyl), m. 265°. Most of the quaternary salts are powerfully antibacterial and V affords protection to mice against *Streptococcus pyogenes*. Certain of them possess a powerful curative action for *Trypanosoma congolense* infections in mice and, consequently, it can no longer be postulated that the presence of an NH<sub>2</sub> group in the biphenyl portion of the mol. is essential to trypanocidal activity. The toxicity and activity of these salts are influenced by the length of the o-alkyl group, the most active member being IV. Although the 3-NH<sub>2</sub> were more active than the corresponding 8-NH<sub>2</sub> salts, the 3-MeO were much less active than the 8-MeO salts.

AN 1950:27413 CAPLUS

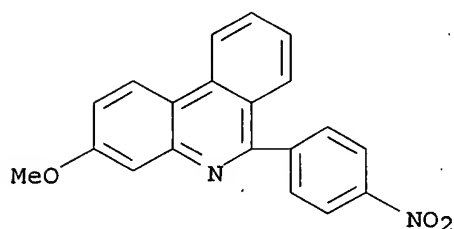
DN 44:27413

OREF 44:5365a-i,5366a-g

TI Potential trypanocides of the N-heterocyclic series. III. Alkoxy- and

## hydroxyphenanthridinium salts

AU Copp, F. C.; Walls, L. P.  
 CS Wellcome Research Labs., Beckenham, UK  
 SO Journal of the Chemical Society (1950) 311-17  
 CODEN: JCSOA9; ISSN: 0368-1769  
 DT Journal  
 LA Unavailable  
 IT 98351-85-0P, Phenanthridine, 3-methoxy-6-(p-nitrophenyl)-  
 RL: PREP (Preparation)  
 (preparation of)  
 RN 98351-85-0 CAPLUS  
 CN Phenanthridine, 3-methoxy-6-(4-nitrophenyl)- (9CI) (CA INDEX NAME)



L6 ANSWER 12 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN  
 AB A solution of 56 g. o-(MeO)2C6H4 in 150 cc. EtOH was stirred and treated alternately with 100 g. iodine and 60 g. HgO during 3 h. After 1 h. the mixture was filtered and the EtOH was distilled from the filtrate. The residue was dissolved in Et2O, washed with Na2S2O3, NaOH, and H2O. After drying, the Et2O was removed and the residue distilled to give 60 g. 4-iodoveratrole (I), b26 163-4°. Heating of 25 g. I and 25 g. Cu powder in CO2 at 235° for 1 h., cooling, extraction with MeOH, and evaporation of the MeOH gave 10 g. 3,3',4,4'-tetramethoxybiphenyl (II), m. 133° (MeOH). II (5.5 g.) in 60 cc. AcOH at room temperature was treated with 1.2 g. HNO3 in 3 cc. AcOH during 15 min. After 30 min., the mixture was warmed on the H2O bath 15 min., cooled, and diluted with ice-H2O. The precipitate was recrystd. from EtOH to give 5 g. 2-nitro-4,4',5,5'-tetramethoxybiphenyl (III), yellow rhombs, m. 149°. Nitration of II or III in AcOH with HNO3 yielded 2,2'-dinitro-4,4',5,5'-tetramethoxybiphenyl (IV), fine yellow needles, m. 218° (EtOH). Hydrogenation of 5 g. III in 200 cc. hot EtOH at ordinary pressure in the presence of Raney Ni yielded 100% 2-amino-4,4',5,5'-tetramethoxybiphenyl (V), colorless prisms from C6H6, m. 151°; piperonylidene derivative, from V and piperonal, yellow plates from EtOH, m. 155°; salicylidene derivative, orange needles from EtOH, m. 144°. Acetylating of V with Ac2O yielded 2-acetamido-4,4',5,5'-tetramethoxybiphenyl (VI), colorless prisms from aqueous EtOH, m. 164°. Similarly (EtCO)2O yielded 2-propionamido-4,4',5,5'-tetramethoxybiphenyl (VII), colorless plates from EtOH, m. 138°, and BzCl in pyridine gave 2-benzamido-4,4',5,5'-tetramethoxybiphenyl (VIII), colorless needles from aqueous EtOH, m. 128°. VI (4 g.) was warmed with 10 cc. POCl3 under reflux 1 h. and the excess POCl3 was removed. The residue was warmed with dilute NaOH, and the precipitate recrystd. from C6H6 to give 85% 6-methyl-2,3,8,9-tetramethoxyphenanthridine (IX), colorless rods, m. 212°; methiodide, from IX and MeI 2 h. at 100°, yellow needles, m. 260-84° (decomposition). In like manner VII yielded 85% 6-ethyl-2,3,8,9-tetramethoxyphenanthridine, colorless prisms, m. 202° (methiodide, yellow needles from dilute EtOH, m. 286°

(decomposition)), and VIII yielded 90% 6-phenyl-2,3,8,9-tetramethoxyphenanthridine, colorless blades, m. 207° (methiodide, yellow needles from dilute EtOH, 273° (decomposition)). A mixture of 10 g. IX, 50 cc. EtOH, and 20 cc. 40% HCHO was refluxed 3 h., and then 20 cc. HCHO was added. After refluxing for another 10 h., 10 cc. HCHO was added, refluxing was continued 3 h., and all solvent was evaporated. The residue was diluted with H<sub>2</sub>O, treated with excess NH<sub>4</sub>OH, boiled, and cooled. The precipitate, upon recrystn. from C<sub>6</sub>H<sub>6</sub> and C<sub>6</sub>H<sub>6</sub>-EtOH, gave 6.5 g. 6-(2,2'-dihydroxyisopropyl)-2,3,8,9-tetramethoxyphenanthridine (X), colorless prisms, m. 214°. X (4.5 g.), 50 cc. H<sub>2</sub>O, and 1.2 cc. H<sub>2</sub>SO<sub>4</sub> were heated on the H<sub>2</sub>O bath and treated with a solution of 7.5 g. K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and 5.6 cc. H<sub>2</sub>SO<sub>4</sub> in 35 cc. H<sub>2</sub>O during 30 min. After 3 h. the mixture was cooled and diluted, and the precipitate was purified by dissolving in NaOH and reprecipitating with AcOH to give 3.9 g. 2,3,8,9-tetramethoxy-6-phenanthridinecarboxylic acid, decomposing 240°, which rapidly decarboxylated when maintained at 245° to yield 2.5 g. 2,3,8,9-tetramethoxyphenanthridine as colorless needles from MeOH, m. 185° (after partially m. 135° and resolidifying); methiodide (XI), pale yellow needles from aqueous EtOH, m. 295°. V (2 g.) and 15 cc. HCO<sub>2</sub>H were refluxed 3 h., cooled, and diluted. The precipitate was recrystd. from aqueous EtOH to give

1.7 g. 2-formamido-4,4',5,5'-tetramethoxybiphenyl (XII), colorless plates, m. 168°. Boiling XII with POCl<sub>3</sub> gave only tar, and reactions at lower temps. or in a solvent gave either a tar or no reaction. Cyclization of 7.5 g. XII by boiling with 10 g. P<sub>2</sub>O<sub>5</sub> in 80 cc. C<sub>6</sub>H<sub>4</sub>Me<sub>2</sub>, followed by methylating with excess MeI at 100°, yielded 0.3 g. XI. The phenanthridine derivs. prepared above all show marked blue fluorescence in neutral solution and stronger fluorescence in AcOH. The yellow methiodides are strongly fluorescent in aqueous EtOH.

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DN 40:5229

OREF 40:876a-i

TI Phenanthridine series. I. Cyclization of 2-formamidobiphenyls

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DT Journal

LA Unavailable

IT 861002-27-9P, Phenanthridine, 2,3,8,9-tetramethoxy-6-phenyl-

RL: PREP (Preparation)

(preparation of)

RN 861002-27-9 CAPLUS

CN Phenanthridine, 2,3,8,9-tetramethoxy-6-phenyl- (CA INDEX NAME)

